

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT(S): L. M. Cirjak, et al.) PATENT APPLICATION
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APPLICATION NO.: 09/981,454 [6353]) Group Art Unit: 1621
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FILED: October 27, 2001) Examiner:
) Michael L. Shippen
)
FOR: Fluid Bed Process for the) Attorney Docket No.:
Acetoxylation of Ethylene in the) BP7339.05
Production of Vinyl Acetate)

Declaration of Bruce Williams Under 37 C.F.R. §1.132

Commissioner
for Patents
Washington, D.C. 20231

I, Bruce Williams, declare as follows:

1. I am currently employed by BP Chemicals Ltd. at the BP Chemicals Research and Technology Centre located at Hull, UK as a Technical specialist. I have been employed by BP Chemicals for 20 years in a professional research capacity.
2. I received a B.Sc. degree from Exeter University, England in Mathematics and Chemistry in 1978, and a Ph.D. degree from Exeter University, England in Organometallic Chemistry in 1981.
3. As part of my professional capacity, I have had 6 years experience in processes to oxidize alkenes, such as production of vinyl acetate from ethylene. In addition I have had a further 9 years experience in the development and scale up of chemical processes such as the carbonylation of methanol to acetic acid.

4. In a gas-phase, fixed-bed oxidation process to form vinyl acetate, a mixture of gaseous ethylene, acetic acid, and oxygen is passed through a bed of solid catalyst particles. Since heat removal is a major technical problem in fixed bed oxidation systems, conventionally catalyst is contained in multiple metal tubes, which are cooled externally.

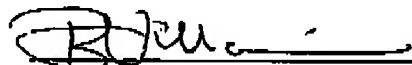
5. In a gas-phase system, care must be taken to avoid mixtures of oxygen, acetic acid, and ethylene that fall within recognized explosion hazard concentrations. In a fixed bed process utilizing a multi-tubular reactor, this is best carried out by pre-mixing the feed gas. In this way the gas composition can be carefully monitored and controlled to avoid mixtures that fall within recognized hazardous regions. This avoids the problems of control and safety that could arise if oxygen were to be fed individually to all the catalyst tubes. In particular, it avoids the problem of hotspots at the point of oxygen introduction to individual tubes. In addition, it prevents the accumulation of a potentially dangerous high concentration of oxygen in a particular area should an individual tube containing catalyst become blocked. Since heat removal is critical to the operation of a fixed bed system, in my opinion, normal chemical engineering practice would avoid such a separate oxygen introduction because accelerated oxidation could cause potential dangerous overheating and catalyst destruction.

6. I have read Calcagno et al., U.S. Patent 3,714,237, which describes catalytic production of vinyl acetate by oxidation of ethylene in acetic acid. This patent further describes that an oxygen-containing stream may be introduced separately from an ethylene-containing stream. Calcagno et al. describes only liquid phase catalytic oxidation reaction. Heat transfer within a liquid-phase system will be better than in a gas-phase, fixed-bed system since the liquid will act a heat transfer medium. Further, oxygen must diffuse through the liquid to react with the ethylene and acetic acid. Since the liquid will act as a quench, this is inherently a safer system.

7. In my opinion, normal chemical engineering practice would not assume that methods of introducing an oxidation agent demonstrated in a liquid phase would be safe or operable in a gas-phase system. This is due to the inherent differences between gas-phase and liquid-phase systems relating to heat removal and formation of an explosive mixture.

8. In a gas-phase oxidation process, normal chemical engineering practice would calculate, from known data, ranges of explosive gas-phase compositions and would set operational control parameters based on those calculated explosive compositions. In practice, control parameters would not be set precisely at the calculated explosive limits, but would be set with a safety margin or danger zone away from the calculated explosive composition. Based on conditions such as control precision, assumed measurement errors, and process variability, a chemical engineer would establish a hazard or danger zone outside a calculated explosive composition. Thus, a chemical engineer would establish the control or set points in the process to be outside that danger zone.

9. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the above referenced application or any patent issuing thereon.



Bruce Williams

Date: 22nd APRIL 2002